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IS 4518-1 (1967): Methods of test for styrene-butadiene
rubbers(SBR) Part I Determination of volatile matter, total
ash, organic acid, soap, antioxidants, bound styrene and
mooney viscosity [PCD 13: Rubber and Rubber Products]
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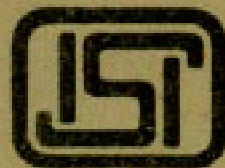BLANK PAGE

IS : 4518 (Part I) - 1967

Indian Standard
METHODS OF TESTS FOR
STYRENE-BUTADIENE RUBBERS (SBR)

PART I DETERMINATION OF VOLATILE MATTER, TOTAL ASH, ORGANIC ACID, SOAP, ANTIOXIDANTS, BOUND STYRENE AND MOONEY VISCOSITY

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PART I DETERMINATION OF VOLATILE MATTER, TOTAL ASH, ORGANIC ACID, SOAP, ANTIOXIDANTS, BOUND STYRENE AND MOONEY VISCOSITY

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Indian Standard
METHODS OF TESTS FOR
STYRENE-BUTADIENE RUBBERS (SBR)

PART I DETERMINATION OF VOLATILE MATTER, TOTAL ASH, ORGANIC ACID, SOAP, ANTIOXIDANTS, BOUND STYRENE AND MOONEY VISCOSITY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 22 June 1967, after the draft finalized by the Rubber Sectional Committee had been approved by the Chemical Division Council.

0.2 The methods of testing styrene-butadiene rubber do not in all cases agree with the methods used for testing natural rubber. It has, therefore, been felt necessary to issue separate standards for methods of test for styrene-butadiene rubbers. In order to make these methods of test available as early as possible, only certain tests are being provided at present, but work is in progress for the preparation of other test methods.

0.3 The methods prescribed in this standard take into consideration the recommendations of International Synthetic Rubber Producers Association. Besides, assistance has also been derived from ASTM Designation D : 1416-1962aT ' Chemical analysis of synthetic elastomers (solid styrene-butadiene copolymers) ' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes the methods of preparing sample and tests for raw styrene-butadiene rubbers for their volatile matter content (SBR : 1 and SBR : 2), total ash content (SBR : 3), organic acid content (SBR : 4), soap content (SBR : 5), antioxidants (SBR : 6), bound styrene content (SBR : 7), and Mooney viscosity (SBR : 8).

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

> NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

3. METHOD OF TAKING OUT TEST PORTIONS FROM SAMPLE BALES

3.1 Take out piece or pieces of rubber from the selected bale by cuts through the entire bale perpendicular to the surfaces of largest area of the bale. The total weight of the material shall be between 600 g and 1 500 g depending on the tests to be done. Using a tool similar to that shown in Fig. 1, obtain plugs of rubber by cutting through the bale. Do not use any lubricant with the tool to obtain piece or pieces of the material. If the material is not tested immediately, place it in a narrow air-tight container of not more than twice the volume of the material or keep it wrapped in two layers of aluminium foil until required.
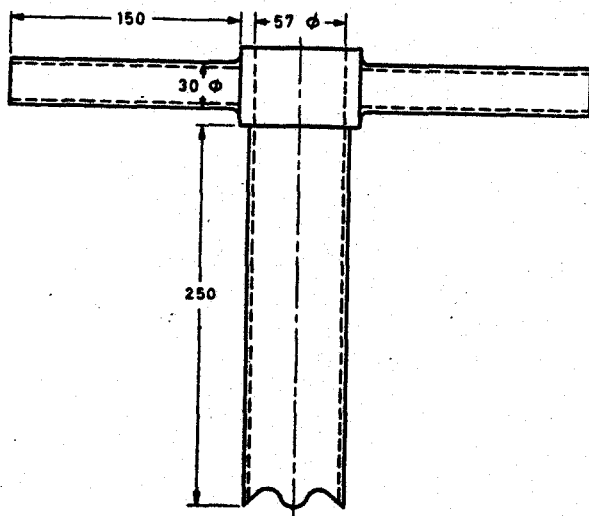
All dimensions in millimetres.

FIG. 1   SAMPLING TOOL

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*Specification for water, distilled quality (*revised*).

3.2 Determine volatile matter on a portion of about 450 g taken from the test portion specified in **3.1**. Retain the dried test portion for chemical tests.

3.3 Homogenization — For the determination of Mooney viscosity, take a test portion of about, but not more than 250 g and mass it by passing 10 times between the rolls of the laboratory mill with rolls having 15 ± 0.05 cm diameter with a working distance between the guides at the nip of 25 to 28 cm. The speed of the slow roll shall be 24 ± 0.5 rev/min and the gear ratio between $1 : 1$ and $1.5 : 1$. Set the distance between the rolls at 1.30 ± 0.15 mm as determined by a lead strip and maintain the roll surface temperature at $50° \pm 5°C$. In passes second to ninth inclusive, the test portion should be doubled upon itself and on the tenth pass it should be sheeted without doubling. Do not allow the test portion to band on the mill rolls and return any solid matter parting from the rubber to it. Cut out disks 4·5 cm in diameter from the thickest part of the sheet and allow them to rest at room temperature for at least 30 minutes before carrying out the Mooney viscosity test.

3.4 Retain the rest of the test portion not required for Mooney viscosity test for testing the vulcanizate characteristics.

DETERMINATION OF VOLATILE MATTER — MILL METHOD (SBR : 1)

1. SCOPE

1.1 This method covers a test procedure for the determination of volatile matter content of styrene-butadiene rubbers which are not too tacky.

2. PRINCIPLE OF THE METHOD

2.1 A test portion of rubber as described in **3.1** (*see* page 5) under method of taking out test portions from sample bales is weighed and passed through heated laboratory mill for a specified time and the loss in weight is determined.

3. APPARATUS

3.1 Chemical Balance

3.2 Laboratory Mill

4. PROCEDURE

4.1 Weigh at least 450 g of material as in **3.1** (*see* page 5) to the nearest 0·1 g. Pass the weighed material repeatedly through a laboratory mill, with the rolls of the mill maintained at 100° to 105°C, and the distance between the rolls at 0·25 ± 0·05 mm. Do not allow the material to band, and take care to prevent any loss of material. At the end of 4 minutes, weigh the sample to the nearest 0·1 g. Pass the material through the mill for an additional 2 minutes and reweigh it. If the weights at the end of the 4 and 6-minute periods are within 0·1 g, calculate the volatile matter; if not, continue passing the material through the mill for 2-minute periods until the weight remains constant within 0·1 g.

5. CALCULATION

5.1 Calculate the percentage of volatile matter as follows:

$$\text{Volatile matter, percent by weight} = \frac{W_1 - W_2}{W_1} \times 100$$

where

W_1 = weight in g of the original material, and

W_2 = weight in g of the material after drying.

DETERMINATION OF VOLATILE MATTER — MILL-OVEN METHOD (SBR : 2)

1. SCOPE

1.1 This method covers a test procedure for the determination of volatile matters in styrene-butadiene rubbers that are too tacky to be handled satisfactorily on a heated mill.

2. OUTLINE OF THE METHOD

2.1 A weighed portion of the rubber is sheeted out on a laboratory mill, then placed in an oven and dried to constant weight. The difference in weight before and after drying is calculated as volatile matter.

3. APPARATUS

3.1 Air-Oven — An air-oven with provision for slow circulation of air in the oven of not less than three changes and not more than ten changes per hour. The temperature of the oven is thermostatically controlled. Provision is made for placing the thermometer near the centre of test pieces to record the actual drying temperature.

3.2 Chemical Balance

3.3 Laboratory Mill

4. PROCEDURE

4.1 Take a portion of rubber at least 250 g prepared according to **3.1** (*see* page 5) and sheet it out on a laboratory mill by pressing it, not more than twice, between the cold rolls set at 0·25 mm distance. Weigh accurately two pieces of about 10 g of sheeted rubber. Place in an oven at 100° to 105°C for at least one hour and until the loss in weight on successive weighings at half-hour intervals is less than 1 mg.

> NOTE — When the sheeting procedure is impracticable, cut the material into pieces larger than 2 mm.

5. CALCULATION

5.1 Calculate volatile matter content as follows:

$$\text{Volatile matter, percent by weight} = \frac{W_1 - W_2}{W_1} \times 100$$

where

W_1 = weight in g of the material before placing in the oven, and

W_2 = weight in g of the material after drying.

DETERMINATION OF TOTAL ASH
(SBR : 3)

1. SCOPE

1.1 This method covers a test procedure for the determination of total ash in styrene-butadiene rubbers.

2. OUTLINE OF THE METHOD

2.1 About 2 g of the material drawn according to **3.1** (*see* page 5) is accurately weighed in a crucible and ignited in a muffle furnace at about 550° ± 25°C until all the carbonaceous material is oxidized. The crucible is then allowed to cool in a desiccator, weighed, and the percentage of ash is calculated. In the case of alum coagulated rubbers which tend to boil over during ashing, ashing is done after wrapping the sample tightly in an ashless filter paper.

3. APPARATUS

3.1 Asbestos Board — approximately 10 cm square and 0·5 cm thick with a hole in the centre to support the crucible so that approximately two-thirds of its depth protrudes below the board.

3.2 Ashless Filter Paper — about 15 cm in diameter.

3.3. Chemical Balance

3.4 Crucible — silica or porcelain crucible having a minimum capacity of 85 ml.

3.5 Desiccator

3.6 Muffle Furnace — with pyrometer and thermostatic control.

4. PROCEDURE

4.1 Heat the crucible to 550° ± 25°C for 30 minutes. Cool in a desiccator and weigh to the nearest 0·001 g. Dry the material in accordance with **4.1** of SBR : 2. Weigh accurately about 3 g of the dried material into the ignited and weighed crucible and rest this in the hole of the asbestos board. Heat gently over a small flame so that the rubber does not ignite and no spurting occurs. When the rubbers completely decompose to a charred mass, transfer the crucible to a muffle furnace at a temperature of 550° ± 25°C. Continue the heating until the ash is free from specks of carbon and then cool in a desiccator and weigh. Replace the crucible in the furnace for 20 minutes, cool in a desiccator and reweigh. Repeat

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heating, cooling and weighing until the weight remains constant within 1 mg.

4.1.1 In the case of alum coagulated rubbers which tend to boil over during ashing, wrap the sample tightly in an ashless filter paper and then proceed as in **4.1**.

5. CALCULATION

5.1 Calculate percentage of total ash as follows:

$$\text{Total ash, percent by weight} = \frac{W_1 - W_2}{W_3 - W_2} \times 100$$

where

W_1 = weight in g of the crucible plus the ash,

W_2 = weight in g of the crucible, and

W_3 = weight in g of the crucible plus the dry material.

DETERMINATION OF ORGANIC ACID CONTENT
(SBR : 4)

1. SCOPE

1.1 This method covers a test procedure for the determination of organic acid content in styrene butadiene rubbers.

2. OUTLINE OF THE METHOD

2.1 Accurately weighed thin narrow strips of dry rubber are extracted with hot ethanol-toluene azeotrope (ETA). The solvent used for extraction as well as the rinsings of the sample are transferred to a volumetric flask and made to a known volume. A made-up portion of the solution is titrated to the first colour change with sodium hydroxide using *m*-cresol purple as indicator. The titrations and weights are used to calculate the organic acid.

3. APPARATUS

3.1 Chemical Balance

3.2 Conical Flasks — 250 ml capacity.

3.3 Graduated Pipette — 25 ml capacity.

3.4 Hot Plate

3.5 Pipette — 100 ml capacity.

3.6 Roll Mill

3.7 Volumetric Flask — 250 ml capacity.

3.8 Wide Mouth Flask — 400 to 500 ml capacity.

4. REAGENTS

4.1 Ethanol-Toluene Azeotrope (ETA) — Mix 70 volumes of ethyl alcohol and 30 volumes of toluene; reflux the mixture for 4 hours over freshly calcined calcium oxide and distil. Discard the first and last fraction keeping only that distillate coming over within a range of 1 deg C. Distilling may be avoided if absolute alcohol is used.

4.2 *m*-Cresol Purple — Indicator solution (0·1 percent) in ethyl alcohol or in water. Neutralize each 0·1 g of indicator with 26·2 ml of 0·01 N sodium hydroxide solution.

4.3 Sodium Hydroxide Solution — 0·1 N.

5. PROCEDURE

5.1 Test Pieces — Dry the rubber in accordance with **4.1** of SBR : 2, pass through mill and get thin (less than 0·5 mm thick) sheet out. Cut the sheet into small strips of not wider than 10 mm and not longer than 50 mm.

5.2 Add 10 ml of ethanol-toluene azeotrope to the wide mouth flask. Accurately weigh 6 g of the strips. Add each strip of the weighed sample separately to the flask, swirling the flask after each addition so that each strip is thoroughly wetted with solvent. To prevent the strips from sticking to the flask, place a filter paper at the bottom of the flask. Use wire gauze or asbestos mat between the flask and the hot plate. Cover the mouth of the flask with an evaporating dish filled with water or use other cooling devices to prevent much loss of solvent and gently reflux for one hour. Decant the liquid into the volumetric flask. Add a second 100-ml portion of the ETA to the rubber sample and reflux again for one hour. Decant liquid into the volumetric flask. Rinse the sample with successive 10 ml portions of fresh ETA and add these rinsings to the volumetric flask. Cool the ETA solution to room temperature and add enough fresh ETA to bring the volume to the mark. Mix the contents thoroughly.

5.3 Pipette out 100-ml portions of ETA solution into 250-ml conical flasks. Add to the above solution 6 drops of *m*-cresol purple indicator solution. Titrate the ETA solution with sodium hydroxide solution to the first colour change. Run a blank titration using 100 ml of fresh ETA that has been treated in the same manner as the sample, and using the same indicator. Deduct the volume of sodium hydroxide solution used for the blank from that used for the titration.

NOTE — When testing alum coagulated rubbers add 5 ml of water to 95 ml of anhydrous ETA.

6. CALCULATION

6.1 Calculate the organic acid content as follows:

$$\text{Organic acid, percent by weight} = \frac{V \times N \times K \times 2\cdot5}{W}$$

where

V = sodium hydroxide solution in ml used for the titration after correcting for the blank;

N = normality of the sodium hydroxide solution;

K = 28·4 when organic acid is determined as stearic acid,
34·6 when organic acid is determined as rosin acid,
31·5 when organic acid is determined as a 50 : 50 mixture of stearic and rosin acid; and

W = weight in g of the original sample.

NOTE — Factor K is theoretical and does not give the exact percentage since soap is not a single chemical compound.

DETERMINATION OF SOAP CONTENT
(SBR : 5)

1. SCOPE

1.1 This method covers test procedure for the determination of soap content in styrene-butadiene rubbers.

2. OUTLINE OF THE METHOD

2.1 Rubber is extracted with ethanol-toluene azeotrope (ETA). A known quantity of the solvent extract is titrated with standard hydrochloric acid, using *m*-cresol purple as indicator, to the first colour change. From the weight of the strips of the material extracted and the titrations, the percentage of soap is calculated.

3. APPARATUS

3.1 Conical Flasks — 250 ml capacity.

3.2 Pipette — 100 ml capacity.

4. REAGENTS

4.1 Hydrochloric Acid — 0·05 N (*see* IS : 265-1962*).

4.2 *m*-Cresol Purple Indicator Solution — 0·1 percent.

5. PROCEDURE

5.1 Prepare the ETA extract following the procedure given in **5.1** and **5.2** of SBR : 4. Pipette out 100 ml of ETA extract into a conical flask and add 6 drops of indicator solution. Titrate with hydrochloric acid to the first change in colour.

5.1.1 Run a blank titration, using 100 ml of fresh ETA and using the same amount of indicator. Deduct the volume of the hydrochloric acid solution used for the blank from that used for the titration.

6. CALCULATION

6.1 Calculate soap percentage as follows:

$$\text{Soap, percent by weight} = \frac{V \times N \times K \times 2{\cdot}5}{W}$$

---

*Specification for hydrochloric acid (*revised*).

where

$V =$ hydrochloric acid solution in ml used for titration after correcting for the blank;

$N =$ normality of the hydrochloric acid solution;

$K =$ 30·6 when the organic acid is determined as fatty acid,

36·8 when the organic acid is determined as rosin acid,

35·3 when soap is to be determined as 50 : 50 mixture of potassium stearate and potassium rosinate,

34·5 when soap is to be determined as a 50 : 50 mixture of sodium stearate and potassium rosinate,

33·7 when the soap is to be determined as a 50 : 50 mixture of sodium stearate and sodium rosinate,

38·4 when soap is to be determined as potassium stearate,

36·5 when the soap is unknown; and

$W =$ weight in g of the material taken.

Note — Factor K is theoretical and does not give the exact percentage since soap is not a single chemical compound.

DETERMINATION OF ANTIOXIDANTS
(SBR : 6)

1. SCOPE

1.1 This method covers a test procedure for the determination of antioxidants in styrene-butadiene rubbers by using spectrophotometer.

2. OUTLINE OF THE METHOD

2.1 The weighed material is extracted with ethanol-toluene azeotrope solution. The total extract is made up to a known volume in a volumetric flask and 2 ml of this extract is further diluted to a known volume with a specified spectroscopic solvent. The absorbence of the dilute extract is determined with an ultraviolet spectrophotometer and the antioxidant content calculated therefrom.

3. APPARATUS

3.1 Chemical Balance

3.2 Pipettes — 2 ml and 3 ml.

3.3 Ultraviolet Photoelectric Spectrophotometer

3.4 Volumetric Flasks — 100 ml.

4. REAGENTS

4.1 Ethyl Alcohol, Toluene Azeotrope (ETA) — as in 4.1 of SBR : 4.

4.2 Spectroscopic Solvent — having an optical transmission greater than 90 percent at the wavelength specified for the antioxidant to be determined when measured in a 1 cm thick quartz cell, as shown in Table 1.

5. PROCEDURE

5.1 Prepare ETA extract following the procedure given in 5.1 and 5.2 of SBR : 4. Accurately pipette 2 ml of the ethanol-toluene azeotrope extract into the 100-ml volumetric flask, and make up to the mark with the spectroscopic solvent (see Table 1).

5.2 Determination of Absorptivity — Determine the absorptivity (the ratio of the absorbence of a solution 1 cm thick and the concentration in grams per litre of the solution that is measured) as in 5.2.1 and 5.2.2 below.

TABLE 1 SOLVENTS AND TEST CONDITIONS FOR DETERMINATION OF ANTIOXIDANT IN STYRENE-BUTADIENE RUBBERS

(*Clause* 4.2)

POLYMER	ANTIOXIDANT	SPECTROSCOPIC SOLVENT	WAVE-LENGTH m/u	BACKGROUND CORRECTION PER GRAM OF SAMPLE PER 250 ml
S B R (hot nonpigmented)	Phenylbetanaph-thylamine	Toluene	309 ± 1	0·085
		Methylcyclohexane	309 ± 1	0·115
	Acetone dipheny-lamine reaction product	85 percent methyl-cyclohexane and 15 percent ethanol*	288 ± 1	0·235
	Mixed alkylated diphenylamines	Methylcyclohexane	288 ± 1	0·225
		85 percent methyl-cyclohexane and 15 percent ethanol*	288 ± 1	0·235
S B R (cold nonpigmented)	Phenylbetanaph-thylamine	Toluene	309 ± 1	0·280
		Methylcyclohexane	309 ± 1	0·325
	Acetone dipheny-lamine reaction product	85 percent methyl-cyclohexane and 15 percent ethanol*	288 ± 1	0·495
	Mixed alkylated diphenylamines	Methylcyclohexane	288 ± 1	0·495
		85 percent methyl-cyclohexane and 15 percent ethanol*	288 ± 1	0·495

*Use anhydrous ethanol.

5.2.1 Obtain two or more representative samples of the antioxidant which shall, in the case of acetone diphenylamine reaction product and mixed alkylated diphenylamines, be heated to a temperature at which they can easily flow. Thoroughly mix the antioxidant and prepare a standard solution of it in the proper spectroscopic solvent. The concentration in grams per litre shall be 0·24 for phenyl β-naphthylamine or acetone diphe-nylamine reaction product, and 0·32 for mixed alkylated diphenylamines. Accurately measure three 3·00 ml aliquots of this solution into clean 100-ml volumetric flasks.

NOTE — It is imperative that scrupulously clean glassware be used throughout this procedure.

5.2.2 Make up the solutions to 100 ml with the spectroscopic solvents. Mix each solution thoroughly. Determine the absorbence of the solutions

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at the appropriate wavelength as shown in Table 1, using the solvent as the blank. Make several measurements on each diluted aliquot until a reproducible result is obtained.

5.3 Determination of Absorptivity of Test Solution — With an ultra-violet spectrophotometer using 1-cm matched quartz cells, measure the absorbence of the solution at the wavelength specified in Table 1, for the antioxidant to be determined. Prepare the blank by mixing together the spectroscopic solvent and the ETA in the same ratio as that used in the solution containing the ETA extract of the rubber. If the absorbence of the solution containing the antioxidant is not between 0·4 and 1·0 take a different aliquot of the ETA extract so as to bring the absorbence within the desired range, being sure to use the same proportion of ETA in the blank as in the sample.

6. CALCULATION

6.1 Calculate the percentage antioxidant as follows:

$$\text{Antioxidant, percent by weight} = \frac{2\,500\,A}{W\,V\,\alpha}$$

where

A = measured absorbence,

W = weight in g of the original sample extracted (*see* **5.1** above),

V = volume of extract in ml dissolved in spectroscopic solvent, and

α = absorptivity [*see* **5.2.2** (SBR : 6)].

6.1.1 Calculate the absorptivity as follows:

$$\alpha = \frac{100\,A}{Y\,Z}$$

where

A = measured absorbence (average of three determinations),

Y = concentration of the antioxidant in the standard as g/1 (*see* **5.2.1** above), and

Z = volume of the aliquot in ml taken from the standard solution.

DETERMINATION OF BOUND STYRENE
(SBR : 7)

1. SCOPE

1.1 This method covers a test procedure for the determination of bound styrene content in styrene-butadiene rubbers.

2. PRINCIPLE OF THE METHOD

2.1 Refractive index of dried, pressed rubber changes with styrene content. Bound styrene is calculated by finding out refractive index.

3. APPARATUS

3.1 Aluminium Foil

3.2 Flashlight Bulb or Auto Lamp — not exceeding 3 candle power.

3.3 Flask — 400 ml capacity.

3.4 Glass Test Piece — standard glass piece for checking the adjustment of the refractometer.

3.5 Razor Blade

3.6 Refractometer* — means of measuring refractive index to read to four places of decimal and having a temperature control such that the temperature could be maintained at 25°C.

3.7 Rolled Aluminium Tube — approximately 50 mm long.

3.8 Tissue Paper

3.9 Vacuum Oven

4. REAGENTS

4.1 Ethyl Alcohol Toluene Azeotrope (ETA) — same as in **4.1** of SBR : 4 (*see* page 11).

4.2 α-Bromonaphthalene

5. PROCEDURE

5.1 Dry the polymer in accordance with **4.1** of SBR : 1 (*see* page 7) and sheet it to a thickness of 0·5 mm or less. Cut the sheeted polymer into

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*Abbe Refractometer will be found convenient.

strips approximately 12·5 mm wide and 25·0 mm long. Fasten one strip to each leg of the aluminium or stainless steel spider, thus allowing each portion of rubber to be contacted on all sides by the solvent. Place the spider and strips in the 400-ml flask, into which 60 ml of solvent has been placed. Extract for 1 hour at a temperature at which the solvent boils gently, replace the solution with another 60 ml of solvent and extract for another hour. Remove the spider from the flask, and dry the rubber in the vacuum oven at about 100°C for at least 1 hour. Avoid plastication of the sample by overheating.

NOTE — It is important that the test specimens be extracted and dried thoroughly, since either residual solvent or incompletely extracted materials will result in erroneous readings of the refractive index.

5.2 After the specimens have been thoroughly dried, remove the strips from the spider. Cut 25·0 mm squares from the clean aluminium foil. Press each strip between two of the aluminium squares, with a force of 25 to 135 kgf for each test piece and at a temperature of 100°C, for 3 to 10 minutes. The period of time most suitable for the pressing is that which results in the most distinct line dividing the light and dark portions of the telescope field. Hold the strips at the same pressure for 10 minutes at room temperature, either by circulating cold water through the press platens or by using a press equipped with both cold and hot platens.

5.3 Measure the refractive index with the refractometer in the same manner as that used for measuring the index of solids. Check the adjustment of the refractometer, using the standard glass test piece (*see* Note 1). Circulating water at 25°C through the prism or performing the test in a constant room temperature at 25°C will avoid the necessity of making a temperature correction and tends to give higher accuracy. Illuminate the instrument with the flashlight bulb or an automobile lamp. In order to provide a beam of light at grazing incidence to the prism, slip the tube of rolled aluminium over the bulb. When reading the glass test piece, use a diffused light source obtained by covering the tube with crumpled tissue paper. Place a small drop of α-bromonaphthalene (*see* Note 2) on the polished flat surface of the test piece, then pass it firmly against the measuring prism of the refractometer, with the polished end towards the light.

NOTE 1 — Clean the glass test piece as well as the prism with ethyl alcohol applied with lens paper, both before and after checking the adjustment of the instrument. Do not use acetone, since it attacks the cement holding the prism in place.

NOTE 2 — Use an absolute minimum amount of α-bromonaphthalene, so that when the test piece is pressed against the prism surface, the liquid film flows down to the polished end of the test piece and yet does not form a wedge of liquid between the face of the test piece and the surface of the refractometer prism. Be sure that the surface of the prism is completely free of contact liquid between the test piece and the light source, otherwise a line will be observed at a position different from that corresponding to the index of the glass test piece and false setting will result.

5.4 Adjust the position of the light source to give the most distinct dividing line. Observe the boundary between light and dark portions on the fields of the telescope, setting the compensating drum, which rotates the Amici prisms used for achromatization, at one of the two positions that give a boundary line most nearly free of colour. Test the contact between the surface of the test piece and that of the prism by firmly pressing the two surfaces together with the finger. If there is no displacement of the boundary line during this operation, the position of the slab may be considered to be satisfactory. Approach the setting from a position on which the intersection of the cross-hairs is in the light portion of the field. Make three readings of the refractive index without moving the test piece. Remove the test piece, clean it, and replace it on the prism in the manner described. Make three more readings of the refractive index. If the two sets of readings do not agree, repeat the procedure until agreement is attained. If the mean of these readings on the test piece is different from the marked value, set the scale of the instrument at the marked value, and bring the dividing line into coincidence with the cross-hairs by turning the small screw that is recessed in the outer barrel of the telescope (*see* Note 1). Whenever the scale is adjusted, repeat the operation of checking the instrument. Before removing the glass test piece or disturbing the scale setting, turn the compensator drum to the second position that gives a boundary line free of colour, and note whether the cross-hairs are still set on the boundary line (*see* Note 2).

NOTE 1 — If it is necessary to make this adjustment frequently, there is an imperfect connection permitting relative motion of the prism and the index arm.

NOTE 2 — If the cross-hairs are found to be displaced by more than 0·000 2, they should be returned to the manufacturer for adjustment.

5.5 Remove the tissue paper from the rolled aluminium tube that was placed over the light bulb. Cut the pressed rubber in the aluminium foil in half. Grasp the narrow end of one of the pieces of aluminium foil and peel it off with a quick continuous pull, leaving the rubber adhering to the other piece of foil. In order to obtain a sharp, clean, perpendicular edge, use the razor blade to cut a strip at least 3 mm wide where the rubber is smoothest and completely free from air bubbles. Place the test pieces on the prism of the refractometer with the cut edge away from the observer, perpendicular to the length of the prism face, and at the same position occupied by the polished edge of the glass test piece. Press the test piece firmly against the foil until it wets the glass, leaving no air entrapped between the prism and the test piece near the cut edge (*see* Note).

NOTE — *Caution* — Under no circumstances should the rubber test piece be clamped between the two refractometer prisms as in measurements with liquids. *Attempts to* close the prisms with rubber between them may distort the hinges, loosen the prism mountings, or damage the prisms.

5.6 Circulate water, having a temperature within 2 deg C of room temperature, through the tap housing to the measuring prisms of the refractometer and through a cored brass block of about the same size as the prism housing, mounted over the test piece. Allow at least one minute for the test piece to reach temperature equilibrium after the block has been placed over the test piece and before an index reading is made.

5.7 Adjust the compensator to give as nearly white light as possible, and set the boundary between the light and dark portions of the field on the intersection of the cross-hairs. In reading the line, adjust the compensator drum so that of the two possible points of compensation, the one used with the rubber test piece is the one nearer to that used with the glass test piece. Approach the setting from a position in which the intersection is in the light portion of the field. Make at least three readings, and if there is a difference of more than 0·000 1 between the readings, mount a new strip on the prism and repeat the readings. If average values obtained on different test pieces from the same pressed test piece differ by more than 0·000 2, extract a new portion of the original sample and repeat the measurements. At each reading record the temperature to the nearest 0·1 deg C by means of a thermometer graduated in units of 0·2 deg C or less.

6. CALCULATION

6.1 Calculate the value of the refractive index at 25°C, from the observations, as follows:

$$n_{25} = n_t + 0·000\ 37\ (t - 25)$$

where

n_{25} = refractive index at 25°C, and

n_t = refractive index observed at temperature t.

6.2 Determine the combined styrene content from the refractive index at 25°C, using Table 1, or calculate the bound styrene, as follows:

Bound styrene, per-
cent by weight = $23·50 + 1\ 164\ (n_{25} - 1·534\ 56) - 349\ 7(n_{25} - 1·534\ 56)^2$

TABLE 1 VALUES OF REFRACTIVE INDEX AND PERCENT BOUND STYRENE

(*Clause* 6.2)

REFRACTIVE INDEX n_{25}	0	1	2	3	4	5	6	7	8	9
					Percent Bound Styrene					
1·515						0·05	0·18	0·31	0·44	0·57
1·516	0·70	0·83	0·96	1·09	1·22	1·34	1·47	1·60	1·73	1·86
1·517	1·99	2·12	2·25	2·37	2·50	2·63	2·76	2·89	3·02	3·14
1·518	3·27	3·40	3·53	3·66	3·78	3·91	4·04	4·17	4·29	4·42
1·519	4·55	4·67	4·80	4·93	5·06	5·18	5·31	5·44	5·56	5·69
1·520	5·82	5·94	6·07	6·20	6·32	6·45	6·57	6·70	6·83	6·95
1·521	7·08	7·20	7·33	7·46	7·58	7·71	7·83	7·96	8·08	8·21
1·522	8·33	8·46	8·58	8·71	8·83	8·96	9·08	9·21	9·33	9·46
1·523	9·58	9·71	9·83	9·95	10·08	10·20	10·33	10·45	10·57	10·70
1·524	10·82	10·95	11·07	11·19	11·32	11·44	11·56	11·69	11·81	11·93
1·525	12·06	12·18	12·30	12·43	12·55	12·67	12·79	12·92	13·04	13·16
1·526	13·28	13·41	13·53	13·65	13·77	13·89	14·02	14·14	14·26	14·38
1·527	14·50	14·62	14·75	14·87	14·99	15·11	15·23	15·35	15·47	15·60
1·528	15·72	15·84	15·96	16·08	16·20	16·32	16·44	16·56	16·68	16·80
1·529	16·92	17·04	17·16	17·28	17·40	17·52	17·64	17·76	17·88	18·00
1·530	18·12	18·24	18·36	18·48	18·60	18·72	18·84	18·96	19·08	19·19
1·531	19·31	19·43	19·55	19·67	19·79	19·91	20·03	20·14	20·26	20·38
1·532	20·50	20·62	20·73	20·85	20·97	21·09	21·21	21·32	21·44	21·56
1·533	21·68	21·79	21·91	22·03	22·15	22·26	22·38	22·50	22·61	22·73
1·534	22·85	22·96	23·08	23·20	23·31	23·43	23·55	23·66	23·78	23·90
1·535	24·01	24·13	24·24	24·36	24·47	24·59	24·71	24·82	24·94	25·05
1·536	25·17	25·28	25·40	26·51	25·63	25·74	25·86	25·97	26·09	26·20
1·537	26·32	26·43	26·55	26·66	26·78	26·89	27·00	27·12	27·23	27·35
1·538	27·46	27·58	27·69	27·80	27·92	28·03	28·14	28·26	28·37	28·48
1·539	28·60	28·71	28·82	28·94	29·05	29·16	29·28	29·39	29·50	29·61

1·540	29·73	29·84	29·95	30·06	30·18	30·29	30·40	30·51	30·62	30·74
1·541	30·85	30·96	31·07	31·18	31·30	31·41	31·52	31·63	31·74	31·85
1·542	31·96	32·07	32·19	32·30	32·41	32·52	32·63	32·74	32·85	32·96
1·543	33·07	33·18	33·29	33·40	33·51	33·62	33·73	33·84	33·95	34·06
1·544	34·17	34·28	34·39	34·50	34·61	34·72	34·83	34·94	35·05	35·16
1·545	35·27	35·38	35·48	35·59	35·70	35·81	35·92	36·03	36·14	36·25
1·546	36·35	36·46	36·57	36·68	36·79	36·89	37·00	37·11	37·22	37·33
1·547	37·43	37·54	37·65	37·76	37·86	37·97	38·08	38·19	38·29	38·40
1·548	38·51	38·61	38·72	38·83	38·93	39·04	39·15	39·25	39·36	39·47
1·549	39·57	39·68	39·79	39·89	40·00	40·10	40·21	40·32	40·42	40·53
1·550	40·63	40·74	40·84	40·95	41·05	41·16	41·26	41·37	41·47	41·58
1·551	41·68	41·79	41·89	42·00	42·10	42·21	42·31	42·42	42·52	42·63
1·552	42·73	42·83	42·94	43·04	43·15	43·25	43·35	43·46	43·56	43·66
1·553	43·77	43·87	43·97	44·08	44·18	44·28	44·39	44·49	44·59	44·70
1·554	44·80	44·90	45·00	45·11	45·21	45·31	45·41	45·52	45·62	45·72
1·555	45·82	45·92	46·03	46·13	46·23	46·33	46·43	46·54	46·64	46·74
1·556	46·84	46·94	47·04	47·14	47·25	47·35	47·45	47·55	47·65	47·75
1·557	47·85	47·95	48·05	48·15	48·25	48·35	48·45	48·55	48·65	48·75
1·558	48·85	48·95	49·05	49·15	49·25	49·35	49·45	49·55	49·65	49·75
1·559	49·85	49·95	50·05	50·15	50·25	50·35	50·44	50·54	50·64	50·74
1·560	50·84	50·94	51·04	51·13	51·23	51·33	51·43	51·53	51·63	51·72
1·561	51·82	51·92	52·02	52·11	52·21	52·31	52·41	52·50	52·60	52·70
1·562	52·80	52·89	52·99	53·09	53·18	53·28	53·38	53·47	53·57	53·67
1·563	53·76	53·86	53·96	54·05	54·15	54·25	54·34	54·44	54·53	54·63
1·564	54·73	54·82	54·92	55·01	55·11	55·20	55·30	55·39	55·49	55·58

MOONEY VISCOSITY
(SBR : 8)

1. SCOPE

1.1 This method covers a test procedure for determination of Mooney viscosity of raw styrene-butadiene rubbers with the help of shearing disk viscometer.

2. OUTLINE OF THE METHOD

2.1 The test involves the application of a torque to a metal disk in a cylindrical chamber filled with rubber under specified conditions. A number proportional to the torque necessary to rotate the disk is taken as an index of the viscosity of the rubber.

3. APPARATUS

3.1 The essential parts of the apparatus are a rotor, a hollow cylindrical die, a means for rotating the rotor, a means for indicating the torque required to rotate the rotor, and controls for maintaining the die at a constant temperature (*see* Fig. 1). The rotor and die cavity have the following dimensions:

	mm
Rotor diameter	38·10 ± 0·03
Rotor thickness	5·55 ± 0·03
Die cavity diameter	50·90 ± 0·15
Die cavity depth	10·60 ± 0·03

3.1.1 The die cavity (*see* Fig. 2) should preferably be formed from only two pieces of unplated hardened steel for improved heat transfer and have radial V-grooves on the flat surfaces to retard slipping. The grooves are spaced at 20° intervals, and extend from at least the 7 mm circle to the 47 mm circle; each groove forms a 90° angle in the die surface with the bisector of the angle perpendicular to the surface and is 1·00 ± 0·25 mm wide at the surface.

3.1.2 The die cavity (*see* Fig. 3) may alternatively be formed from four pieces of steel with rectangular-section grooves (*see* Fig. 3A and 3B) on the cavity surfaces to retard slipping. The grooves are 0·80 ± 0·02 mm wide, of uniform depth between 0·30 ± 0·05 mm, and spaced on 1·60 ± 0·04 mm centres. The flat surfaces of the cavity have two sets of these grooves at right angles to each other.
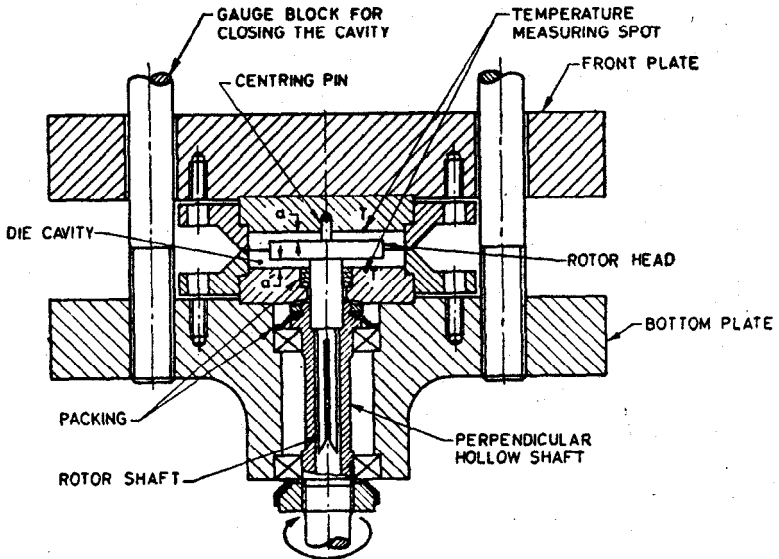
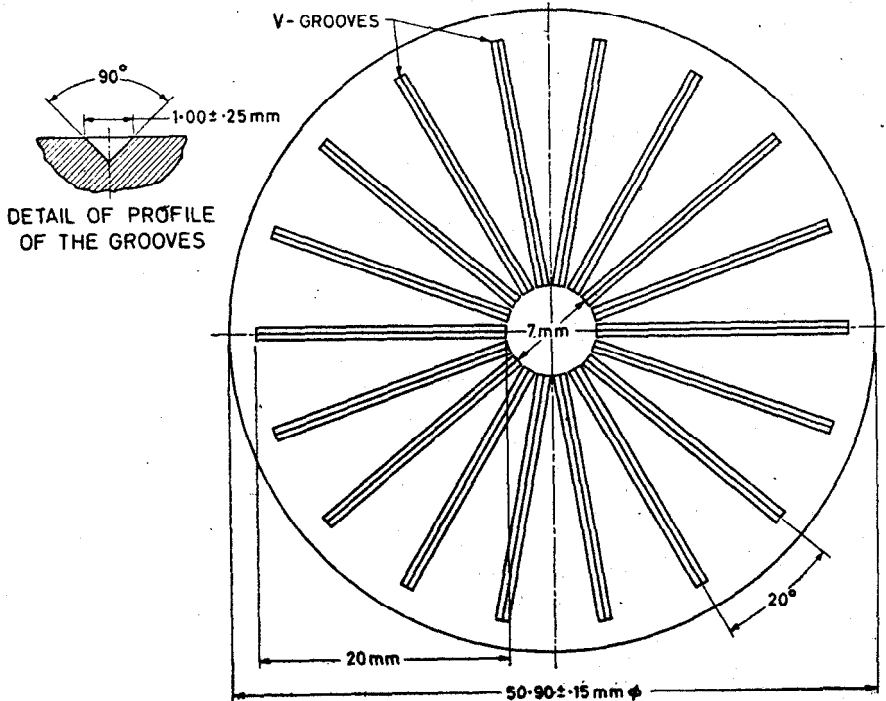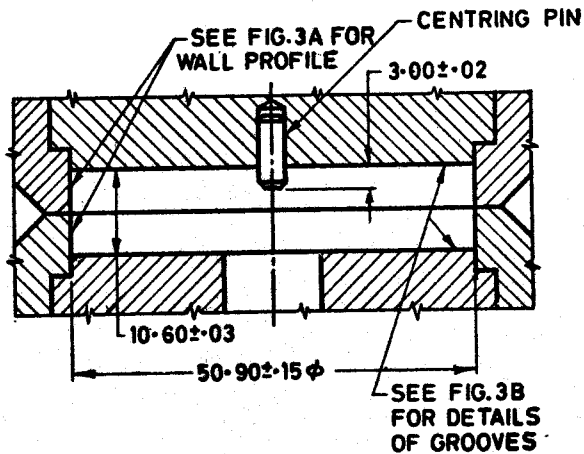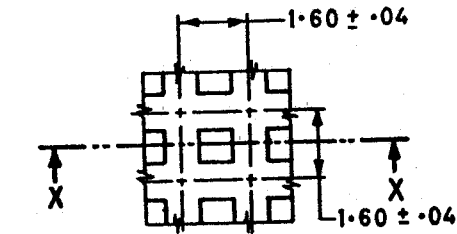FIG. 1   ESSENTIAL PARTS OF THE SHEARING DISK VISCOMETER
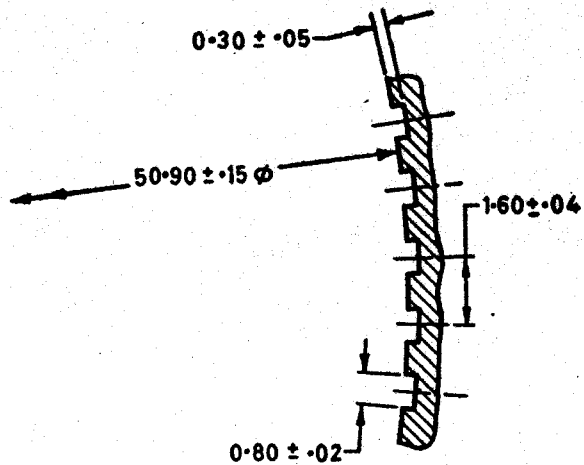

FIG. 2   RADIAL V-GROOVES OF DIE CAVITY

25

1·60 ± ·04

1·60 ± ·04

0·80 ± ·02

0·30 ± ·05

SECTION XX

3A Wall Profile of the Die Cavity

0·30 ± ·05

50·90 ± ·15 ⌀

1·60 ± ·04

0·80 ± ·02

3B Rectangular-Section Grooves

SEE FIG.3A FOR WALL PROFILE

CENTRING PIN

3·00 ± ·02

10·60 ± ·03

50·90 ± ·15 ⌀

SEE FIG.3B FOR DETAILS OF GROOVES

All dimensions in millimetres.

FIG. 3 DIE CAVITY

3.1.3 The rotor (*see* Fig. 4) surfaces are grooved as described for the die cavity formed from four pieces of steel. The hardened rotor is fastened to a shaft not exceeding 11 mm in diameter and positioned securely so that in the closed die cavity the clearance above the rotor (*see a* in Fig. 1) does not differ from the clearance below the rotor (*see a* in Fig. 1) by more than 0·25 mm. The eccentricity or runout of the rotor while turning in the viscometer should not exceed 0·013 mm. The rotor shaft bears on the spindle which turns the rotor and not on the wall of the die cavity. The clearance at the point where the rotor enters the cavity should be small enough to prevent rubber leaving the cavity. A grommet may be used as a seal at this point.
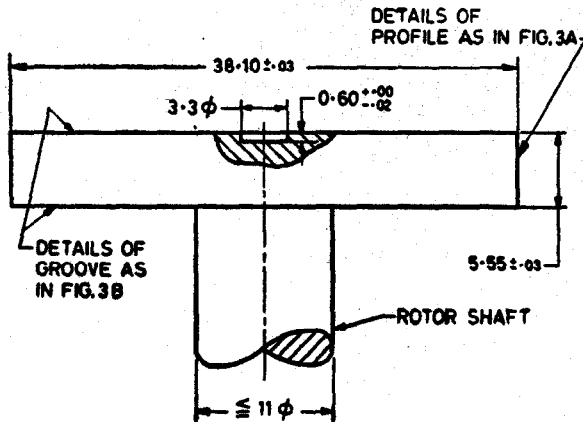
All dimensions in millimetres.

FIG. 4   ROTOR

3.1.4 The dies forming the die cavity are mounted on, or form part of platens equipped with a heating device capable of maintaing the die cavity within 0·5 deg C of the test temperature.

NOTE — The test temperature is defined as the steady-state temperature of the closed cavity, with rotor in place but without rubber. Since a temperature differential exists between the platens and the die cavity, it is necessary to adjust the platen temperatures to obtain the correct cavity temperature. In making such adjustments it is important that the temperature of the two platens be within 0·3 deg C of each other. The cavity temperature may be measured with calibrated thermocouples or thermistors using lead wires about 0·3 mm in diameter to minimize thermal conduction to the exterior.

3.1.5 The die cavity may be closed by hydraulic, pneumatic or mechanical means. If fluid pressure is used, a force of 1 400 kg may be required for the initial closure when rubbers of very high viscosity are tested. At least 10 seconds before starting the viscometer, the force is reduced to 340 ± 20 kgf and maintained at this value during the test. If mechanical closure is used, the platens are adjusted, preferably by means of a gauge

block, so that the total deformation of the parts is between 0·10 mm and 0·15 mm when the die cavity is closed at the test temperature. For all types of closing device, the closure is adjusted so that a piece of thin soft tissue paper not thicker than 0·04 mm, placed between the meeting surfaces shows a continuous pattern of uniform intensity when the die cavity is closed.

3.1.6 The torque required to turn the rotor is recorded or indicated on a linear scale graduated in Mooney viscosity units so that a torque of 84·6 ± 0·2 cm.kg on the rotor equals 100 on the scale. The scale is capable of being read easily to 0·5 unit and is calibrated by means of weights fastened to a special rotor with flexible wire not over 0·5 mm in diameter passing over pulleys free of friction. During the calibration the rotor is turned at 2 rev/min and the platens are at the test temperature. The reading should be zero with the torque removed and 100 ± 0·5 when a torque of 84·6 cm kg is applied.

3.1.7 After calibration, the scale reading is adjusted to zero with the test rotor turning in the empty, closed die cavity at 2 rev/min. The fluctuations in readings during a period of a half minute or more should be less than 0·5 unit.

4. PROCEDURE

4.1 Preparation of Rubber Test Piece — Prepare two disks of rubber each about 45 to 50 mm in diameter and 6 to 8 mm in thickness to fill completely the cavity of the viscometer, in accordance with **3.3** (*see* page 6). The rubber disks should be as free as possible from air and from pockets that may trap air against the rotor and die surfaces. A hole is pierced or cut through the centre of one disk to permit the insertion of the rotor stem.

4.2 Heat the die cavity and rotor to the test temperature of 100°C and allow to reach a steady-state. Open the die cavity, insert the rotor stem through the hole in test disk, place the rotor in the viscometer, place the solid test disk centrally on the rotor and close the die cavity as quickly as possible. Insert, if necessary, a film of transparent cellulosic material approximately 0·03 mm in thickness between the rubber and metal surfaces to facilitate removal after test when testing low-viscosity or sticky materials. Note the time of closing the die cavity and allow the rubber to heat for one minute. Turn the rotor at 2·00 ± 0·02 rev/min unless otherwise specified after the specified time of preheat. If the viscosity is not recorded continuously, observe the indicator and scale continuously during the 30 second interval preceding the specified time of reading and the minimum value to the nearest 0·5 unit during this interval as the viscosity. For reference purposes, take readings at 5 second intervals from 1 minute before to 1 minute after a 4-minutes test period. Draw smooth

curve through the minimum points of the periodic fluctuations or through all points if there are no fluctuations. Take the viscosity as the point where the curve intersects the time specified. If a recorder is used, take the viscosity from the recorded curve in the same manner as specified for the plotted curve.

NOTE — The temperature gradients and rate of heat transfer vary among viscometers, particularly if different types of heating are employed. Therefore, the values obtained with different viscometers may be expected to be more comparable after the rubber has attained the test temperature. Usually this condition is reached about 10 minutes after the die cavity is closed. For most rubbers, the reading is not altered appreciably by permitting the rubber to heat in the viscometer for different times, provided the viscosity is read at a specified time. The running time should never be less than 2 minutes.

5. REPORT

5.1 The report shall include the following:

a) Mooney viscosity number (M),

b) Temperature of test,

c) Rotor size, large or small (L or S),

d) Time interval of preheat, and

e) Time of reading after preheat.

NOTE — Results of a typical test shall be reported as follows:

$$50 \; ML \; 4 \; (\; 100°C \;)$$

where

$M =$ Mooney units,

$L =$ large rotor,

$4 =$ test period in minutes, and

$100°C =$ test temperature.

6. REPRODUCIBILITY OF RESULTS

6.1 Note on Reproducibility of Results — The standard deviation for measurement on the same instrument of a uniform sample of rubber is about 0·2 unit. Variation in sample preparation results in standard deviations of about 1 unit; recorded figures from laboratories working with synthetic rubber production range from 0·20 to 0·75 units. The standard deviation is substantially independent of the viscosity, at least within the range of 40 to 90 units. Variability among laboratories may cause even larger variations in results. Parts of the interlaboratory variability is due to sample preparation and part to errors in calibrating or adjusting the viscometer.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 N = 1 kg.m/s^2$
Energy	joule	J	$1 J = 1 N.m$
Power	watt	W	$1 W = 1 J/s$
Flux	weber	Wb	$1 Wb = 1 V.s$
Flux density	tesla	T	$2 T = 1 Wb/m^2$
Frequency	hertz	Hz	$1 Hz = 1 c/s (s^{-1})$
Electric conductance	siemens	S	$1 S = 1 A/V$
Electromotive force	volt	V	$1 V = 1 W/A$
Pressure, stress	pascal	Pa	$1 Pa = 1 N/m^2$